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ORIGINAL RESEARCH

Texture of Composite Resins Exposed to Two- and Three-Body Wear *in vitro*

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ABSTRACT

Purpose: To analyze on scanning electron microscopy (SEM) pictures from eight composite resins, taken in the centers of the initial, the middle and the terminal thirds of *in vitro* produced wear tracks morphological features to explain causative mechanisms for the material wear observed under two- and three-body wear.

Materials and methods: *In vitro* wear behavior of eight composite resins, three conventional and five nanofiller containing marketed products was evaluated using a custom-made Zr-ball-on-disk sliding device. The composite specimens were subjected to 50,000 one-way sliding cycles (1.2 Hz, 50 N load), either simulating two-body wear with water as the intermediate medium or three-body wear using aqueous suspensions of polymethyl methacrylate (PMMA) beads and poppy seeds, respectively. Volume loss of the materials was determined in previous study. Representative specimens were selected for inspection by scanning electron microscopy at 500-fold magnification. From each of the 24 wear tracks microphotographs were taken in the central deepest parts of the initial, middle and terminal thirds of the tracks.

Results: For most materials morphological differences were detected depending on the location within the wear track. As a rule, the surface deterioration found increased toward the final part of the wear scar. According to common classification in tribology abrasive wear and fatigue wear, or a combination of both mechanisms were found for all materials tested. Wear was dependent both on the testing mode and on the composition of the individual composite resin material.

Conclusion: The morphological assessment of wear tracks reflects the nature of the abrasive and reveals insight into the mechanism generating wear patterns. Morphological details confirmed abrasive and fatigue-related wear as main failure mechanisms. Selection of food-like slurries as third-body media, such as poppy seed suspension is mandatory to simulate wear of composite restorations in occlusal cavities where three-body wear is the dominating determinant of loss of substance and surface deterioration.

Keywords: Two-body wear, Three-body wear, Morphological feature, Composite resin.

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INTRODUCTION

In spite of the recent improvements of composite resin formulations and properties wear of occlusal resin-based restorations is still a matter of concern, especially in case of large restorations or with patients exerting parafunctional habits, such as grinding or clenching.[1] Wear is by definition a progressive loss of substance resulting from mechanical interaction between two contacting surfaces, which are in relative motion.[2] The intraoral tribosystem is highly complex and thus so far not fully simulated in laboratory testing methods.[3,4] Several *in vitro* testing devices have been described in the dental literature,[5,6] however reasonably good correlations with clinical wear data were only reported for few of them.[7,8] With most of the other testing devices product rankings or trends are achieved only; their clinical relevance remains often dubious.

Irrespective of the test methodology used the results of wear tests should be quantifiable and the morphology of the worn surfaces should be described, since morphological details of the worn surfaces can offer valuable clues to understanding of the underlying wear mechanism. In an attempt to evaluate wear characteristics of several composite resins Koottathape et al[9] have recently reported quantitative volume loss data using a ball-on-disk sliding method after 50,000 loading cycles, both under two- and three-body wear conditions. Furthermore, the authors described the texture of the deepest parts of the wear tracks produced. Since sliding wear is a dynamic process the surface morphology of the tracks along the sliding pathway may differ by location and type of composite resin used, as indicated by the non-uniform outline of the wear scars produced.

Therefore, the aim of the present investigation was to analyze on scanning electron microscopy (SEM) pictures, taken at the initial, the middle and the terminal thirds of *in vitro* produced wear tracks morphological features that might explain causative mechanisms for the material wear observed.

MATERIALS AND METHODS

Table 1 shows the composite materials investigated with compositional details. For wear testing specimens were produced in cylindrical alumina molds, 8 mm in diameter and 2 mm in depth. After filling the excess was covered with a Mylar strip, pressed flush and light activated for 40 seconds (Quartz-tungsten-halogen (QTH) unit: XL3000, 3M ESPE, MN, USA; >500 $mWcm^{-2}$). The specimens were stored in 37°C water for 1 week before the composite excess was removed on wet SiC paper, grits 600 through 4000.

The wear-testing device was a custom-made ball-on-disk sliding machine fitted with a ZrO_2-ball (4 mm in diameter), serving as 'antagonist' cusp. The composite surface was loaded (5 N) at 15° angulation, the sliding path length was 3.7 mm, then the ball was lifted and returned to the starting position for the next cycle (1.2 Hz, 50,000 cycles). The specimens (n = 5) were mounted in an aluminum container either filled with water for two-body wear testing or with aqueous slurries of polymethyl methacrylate (PMMA) beads (30 mass%; Palapress, Heraeus Kulzer, Germany, average bead size 40 µm) and lightly preground poppy seed (33 mass%) for three-body wear testing, respectively. The slurries were renewed after each 10,000 cycles. Testing was done at ambient laboratory atmosphere. Quantitative wear was determined as loss of substance with a digital charge coupled device (CCD) microscope (VHX-1000, VH-Z 100R lens, Keyence Corp., Osaka, Japan) at 100-fold magnification at 5 µm steps along the z-axis.

From each composite the sample representing best the average loss of wear of the group was selected, sputtered with Au and inspected with a SEM at 10 kV acceleration (VE-8800, Keyence Corp, Osaka, Japan). Photographs at 500-fold magnification were taken in sliding direction in the centers of the initial, the middle and the terminal thirds of the wear tracks produced during the 50,000 sliding cycles.

RESULTS

Table 2 shows the average loss of volume from 5 specimens after 50,000 sliding wear cycles in water, PMMA suspension and poppy seed, respectively.

Figure 1A displays the morphological features of the microfilled composite DUR after two- and three-body sliding wear contacts. When loaded under water the initial segment of the wear scar looked uniform and free of defects, indicating compression of the material under the load of

Table 1: Materials tested

Material	*Type*	*Code*	*Manufacturer*	*Composition*		
				Monomer	*Filler*	*vol%*
Durafill®VS	microfilled	DUR	Heraeus Kulzer, Hanau, Germany	Bis-GMA, UDMA, TEGDMA	SiO_2 (20–70 nm), prepolymer (<20 µm), SiO_2 in prepolymer	66
Clearfil AP-X	hybrid	APX	Kuraray, Okayama, Japan	Bis-GMA, TEGDMA	Silanated Ba glass, silanated colloidal SiO_2, silanated SiO_2 (0.1–15 µm)	70
Filtek™ Z250	micro-hybrid	Z250	3M ESPE, St. Paul, MN, USA	Bis-GMA, Bis-EMA, UDMA	SiO_2, ZrO_2 (0.01–3.5 µm, average 0.6 µm)	60
Filtek™ Supreme XT	nanofilled	FIL	3M ESPE, St. Paul, MN, USA	Bis-GMA, UDMA, Bis-EMA, TEGDMA	SiO_2/SrO_2 clusters (0.8–1.4 µm), SiO_2 (20 nm)	59.5
GC Kalore	nano-hybrid	KAL	GC Corporation, Tokyo, Japan	UDMA(DuPont), DMA, UDMA	Prepolymer (incl. 400 nm SrO_2 and 100 nm lanthanoid fluoride), F-Al-silicate (700 nm), Sr-Ba-glass (700 nm), SiO_2 (16 nm)	69
MI Flow	nano-hybrid	MFL	GC Corporation, Tokyo, Japan	UDMA, Bis-MEPP, DMA	SiO_2 and Sr-doped nanofiller (700 nm), lanthanoid fluoride (100 nm)	40
Venus®Diamond	nano-hybrid	VED	Heraeus Kulzer, Hanau, Germany	TCD-DI-HEA, UDMA	Ba-Al-F-silicate glass (< 20 µm), SiO_2 (5 nm)	64
Venus®Pearl	nano-hybrid	VEP	Heraeus Kulzer, Hanau, Germany	TCD-DI-HEA, UDMA	Ba-Al-F glass, prepolymerized filler, SiO_2 nanofiller (5 nm - 5 µm)	59

Abbreviation: Bis-GMA: Bisphenol-A diglycidylether methacrylate; Bis-MEPP: 2,2-Bis(4-methacryloxypolyethoxyphenyl) propane; UDMA: urethane - dimethacrylate; TEGDMA: triethylene glycol dimethacrylate; Bis-EMA: Ethoxylatedbisphenol-A dimethacrylate; DMA: Dimethacrylate; TCD-DI-HEA: Bis-(acryloyloxymethyl) tricyclo (5.2.1.02,6) decane

Table 2: Average volume loss of materials after 50,000 wear cycles

Material	*Wear medium*	*Volume loss (mm^3) ± SD*
DUR	Water	0.077 ± 0.016
	PMMA	0.543 ± 0.156
	Poppy seed	1.609 ± 0.296
APX	Water	1.285 ± 0.268
	PMMA	1.381 ± 0.172
	Poppy seed	0.142 ± 0.034
Z250	Water	2.031 ± 0.843
	PMMA	0.489 ± 0.098
	Poppy seed	0.107 ± 0.067
FIL	Water	2.088 ± 0.401
	PMMA	0.433 ± 0.056
	Poppy seed	0.114 ± 0.116
KAL	Water	0.144 ± 0.032
	PMMA	1.462 ± 0.039
	Poppy seed	0.423 ± 0.332
MFL	Water	0.125 ± 0.042
	PMMA	1.202 ± 0.230
	Poppy seed	0.462 ± 0.253
VED	Water	0.962 ± 0.255
	PMMA	2.098 ± 0.293
	Poppy seed	0.356 ± 0.080
VEP	Water	0.685 ± 0.180
	PMMA	2.040 ± 0.145
	Poppy seed	0.114 ± 0.027

Quantitative wear data adapted from Koottathape et al.[9]

the ZrO_2-ball, whereas in the middle and terminal aspects localized defects and cracks were detected, extending in directions perpendicular to the sliding pathway and mostly following the boundaries between prepolymerized particles and the surrounding matrix. In contrast, wearing with PMMA slurry had produced jointed appearance initially and terminally. The middle part of the track is less bumpy with alternating smooth and ragged areas. Parts of the composite are broken out of the surface in every location, however without leaving deep craters. Under the effect of poppy seed abrasion deep destruction and pronounced cracking along and through prepolymer particles was seen.

Under attrition (two-body wear) and PMMA abrasion the hybrid type APX (Fig. 1B) showed asperities of the largest glass fillers protruding from the surface. Especially in the final third of the tracks holes were seen after exfoliation of fillers. Wear with the poppy seed slurry produced similar morphology, although the overall appearance of the surface was smoother.

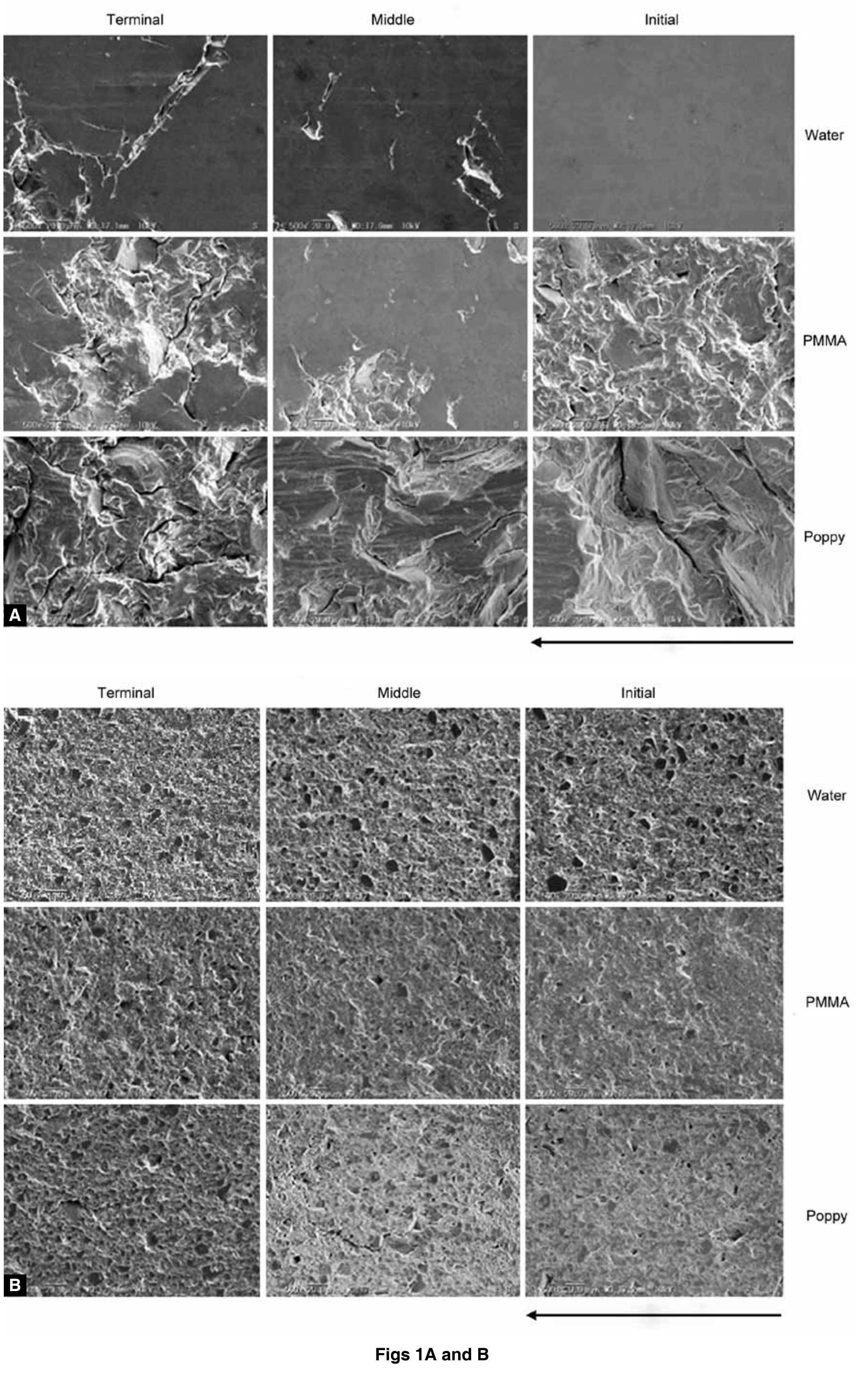
The micro-hybrid composite Z250 (Fig. 1C) appeared rather smooth and uniformly worn under two-body loading. In the initial segment the largest fillers were exposed over the surface, whereas in the terminal segment pit holes were shown after loosening and debonding of the larger spherical filler particles. When worn with the intermediate PMMA slurry, in all three segments of the trace characteristic areas were displayed, showing alternating delamination of parts from the surface and uniformly abraded zones alike. The poppy seed abrasive had apparently only superficially worn the composite, shallow scratches in sliding direction were illustrated in each of the sections investigated.

FIL is a nanofilled composite resin. The SEM investigation (Fig. 1D) revealed a very similar appearance as for Z250, in particular when exposed to two-body wear. In the initial third of the scar black spots, apparently the clustered elements were seen protruding from the surface. In the central and terminal views it seemed that many of the clusters were abraded, almost down to the surrounding filled matrix polymer. Noticeably, the terminal segment showed numerous cracks, perpendicular to the sliding direction. PMMA slurry had produced surface delamination, similar to the effects seen on Z250. Under the action of poppy seed the initial part of the track was somewhat bumpy, the central and terminal parts were rather smooth with superficial scratching left after the abrading poppy seed parts. The circular local defect seen in the terminal picture was apparently caused by a defect or void introduced during specimen preparation.

The nano-hybrid material KAL (Fig. 1E) was seemingly only superficially worn during two-body testing. In the first third the wear track showed striations in sliding direction and dominantly compression of the composite (dark zone). In the middle part the prepolymerized particles were almost as much abraded as the surrounding polymer. The terminal sector showed a uniformly worn material with fillers and matrix in the same level. PMMA had caused pronounced relief polishing, the prepolymers protruded out of the surrounding. Both in the middle and the terminal zone cracks around prepolymer fillers and fractures were identified. For poppy seed wear the first part of the track was very gently abraded, whereas surface cracking and deep fractures were increasingly more dominant in the middle and terminal thirds.

The morphology of the flowable composite MFL (Fig. 1F) was largely comparable with the texture found on KAL. Attritional wear in water was very shallow, no differences could be observed in different sections of the track. With PMMA, however, surfaces were seemingly deeper attacked. The effects increased from the initial to the final aspect. The loosened particles displayed in the terminal view could be interpreted as chipped composite parts, pressed into the surface. Poppy seed had produced rather uniform wear in the initial part, yet extremely broken-up and craggy surfaces in the middle and final thirds. Many of the deep cracks extended perpendicular to the ball sliding motion.

VED (Fig. 1G) is another nano-hybrid composite characterized by the presence of very large glass filler particles. Under attrition wearing already in the initial part of the track deep craters resulting from plucking-out of such


Terminal
Middle
Initial
Water
PMMA
Poppy
A
Terminal
Middle
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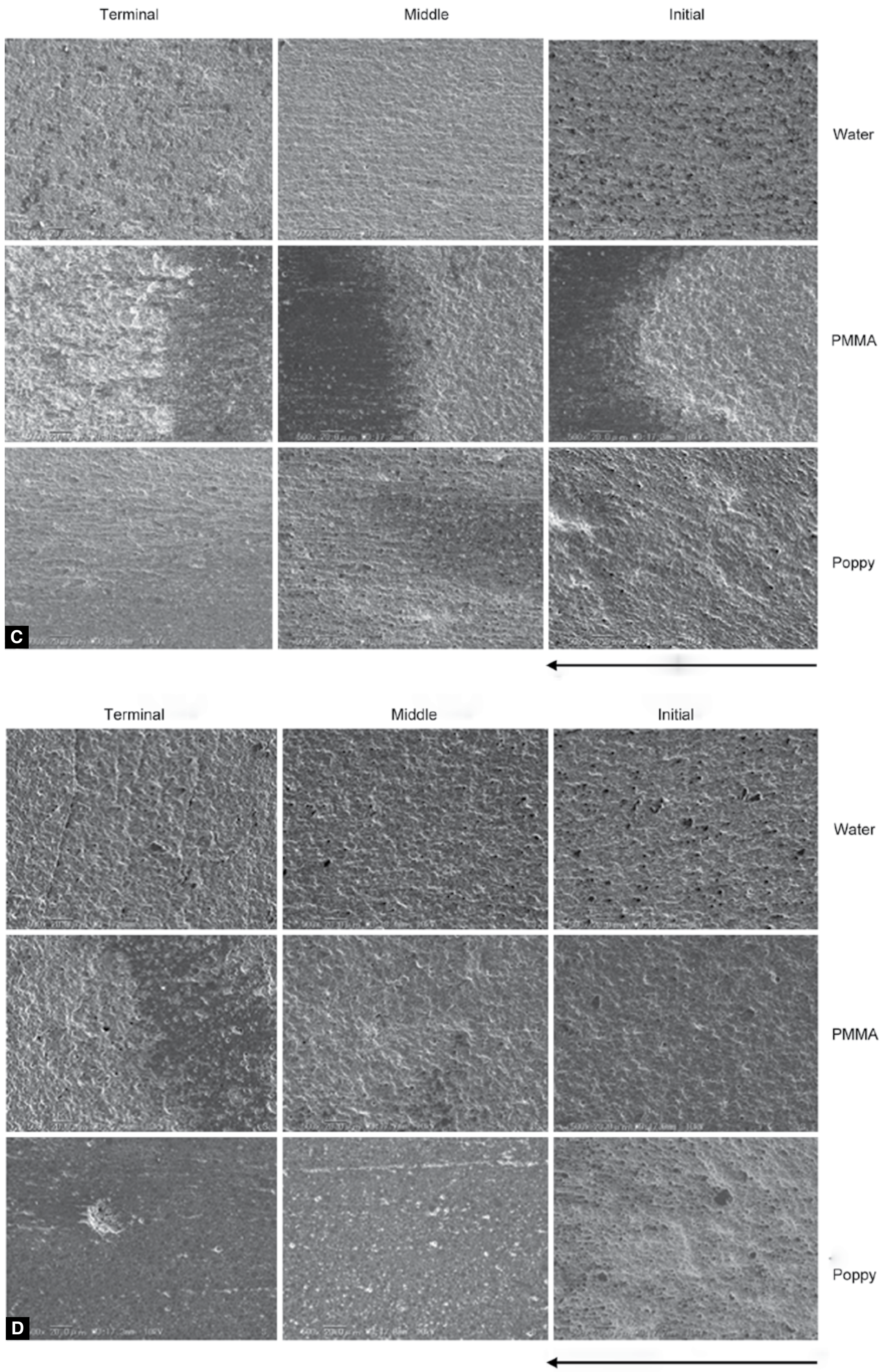
Figs 1A and B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Poppy
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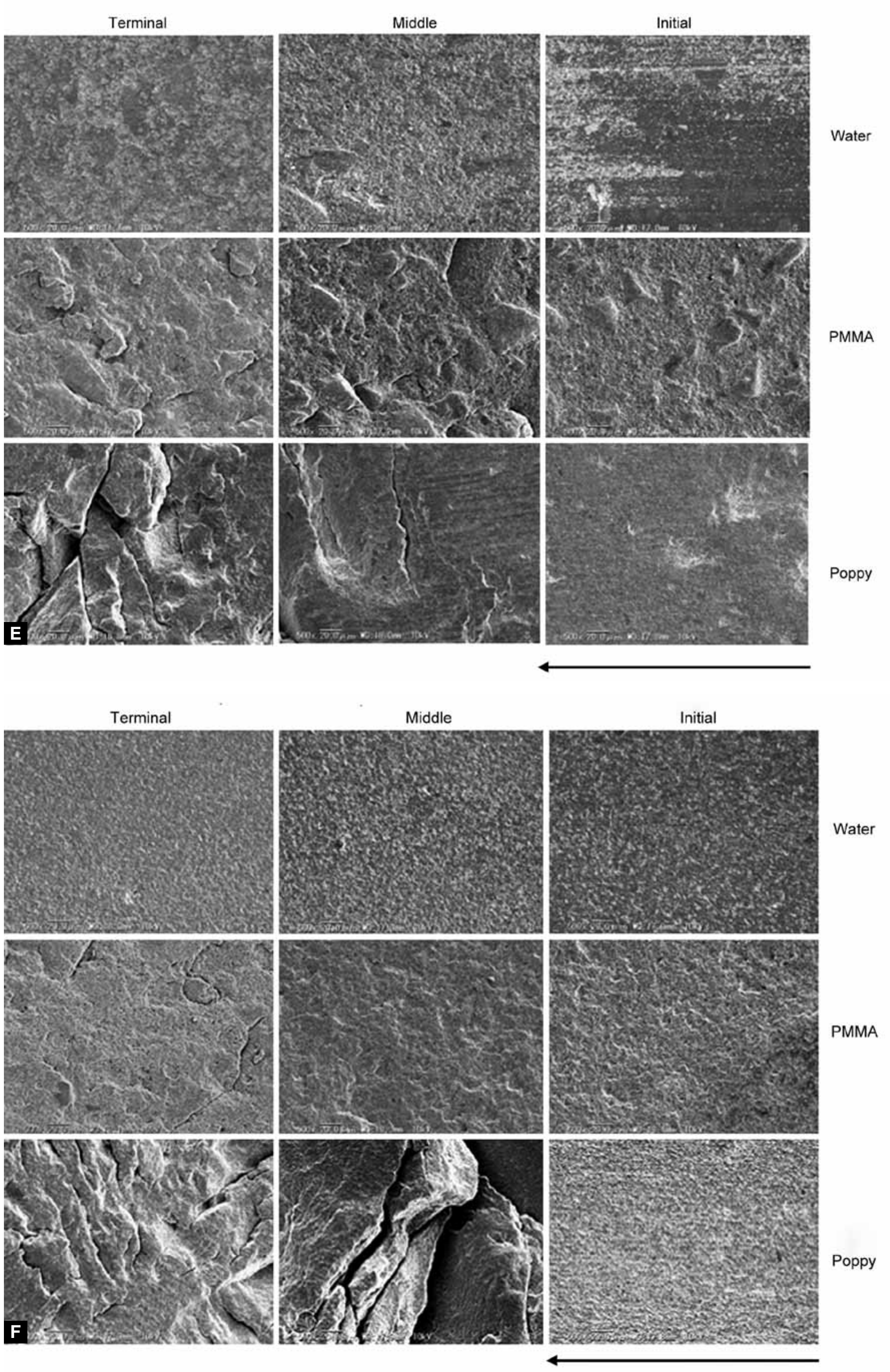
Figs 1C and D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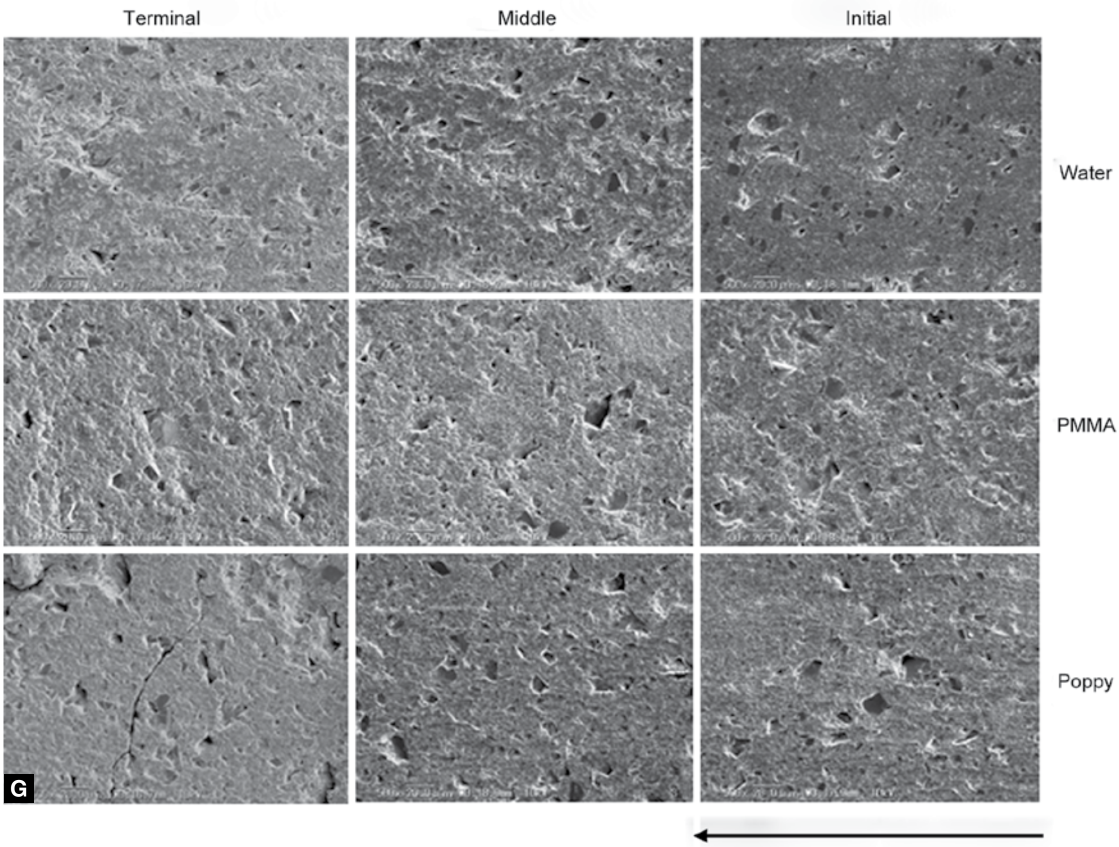
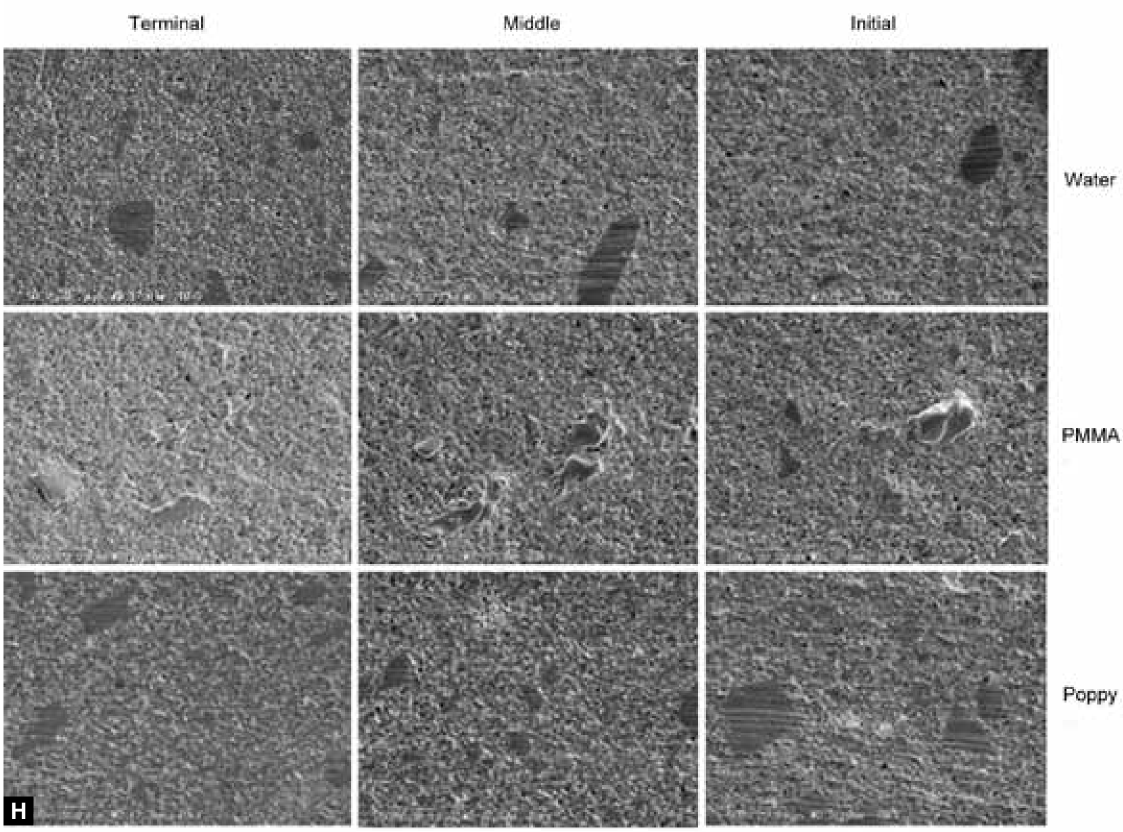
Figs 1E and F





Figs 1G and H

Figs 1A to H: SEM microphotographs (x500) from central deepest parts of the initial, middle and terminal thirds of wear tracks, produced after 50,000 sliding cycles under two-body wear (Water), three-body wear (suspension of PMMA beads) and three-body wear (aqueous suspension of preground Poppy seeds). The arrows indicates the sliding direction of the antagonist ball, (A) DUR (B) APX (C) Z250 (D) FIL (E) KAL (F) MFL (G) VED (H) VEP large fillers were seen, in the further course the composite surface exhibited corrugated rounded-down texture. The PMMA exposed specimen demonstrated similar appearance as the one abraded under water contact, showing numerous holes left after filler pluck-out. The large fillers were also the main target points during poppy seed abrasion. In areas neighboring lost fillers the surface of the matrix loaded with fine-grained filler was mostly smoothly worn. In the terminal part of the wear tracks occasionally long cracks were observed, stretching between and around large fillers or holes left behind.

In Figure 1H the wear characteristics of VEP are illustrated. The characteristic trait of this composite was the large prepolymerized particles that were very uniformly abraded under attrition in the same plane as the surrounding matrix. Fine grooving was recognized along the slide path of the antagonist ball. Under the wearing action of the PMMA beads or fractured parts thereof in all three segments apparently loosened parts of the composite were carried across and remained on the surface, presumably acting as new additional abrasive objects. Under the strain exerted by poppy seeds uniform surface characteristics were found along the entire sliding path, revealing the structural details of this composite resin.

DISCUSSION

In general terms of tribology wear is classified as adhesive wear, abrasive wear, wear caused by fatigue and wear due to environmental factors.[2] The morphological evaluation of the present specimens gave in accordance with previously published research[10-13] evidence for abrasive wear and fatigue-related wear, whereas environmental effects were unlikely, since water was used for the two-body test and aqueous suspensions of the third-body media. However, due to possible fermentation processes during the use of the poppy seed slurry corrosive wear cannot be generally excluded.[14]

This *in vitro* test used the ZrO_2-ball-on-disk model for simulation of composite wear. The material and shape of suitable abraders is still a matter of dispute. Among others, natural enamel, steatite, ceramics or stainless steel have been used as antagonist materials, however none of them qualified as the ideal material for *in vitro* wear testing.[5,15]

One of the limitations of the present study is, that the test design mainly simulates wear of full-coverage composite restorations. In most cases of occlusal restorations the fillings are entirely or partly surrounded by natural enamel, supporting occlusion and articulation. Assuming that composite resin wears more extensively and faster than enamel, direct contact between the opposing cusp and the restoration surface will be lost after short time in service. Thus, even in the occlusal contact area (OCA) no continuous wear will occur unless abrasive slurry, the food bolus, is compressed between the surfaces and dissipated along food shedding pathways in contact-free areas (CFA). With time the distance between the opposing cusp and the restoration becomes wider due to the abrading action of the slurry, and the stress transferred to the restoration surface decreases. According to Pallav et al,[16] even minor variations of the food-film thickness will have considerable impact on wear rates of composite resins. In our testing procedure, each sliding cycle was done under the same load. Therefore, the results may assumedly overemphasize the wear that might be expected in a clinical situation.

Use of a reasonable food-simulating medium is an essential requirement for *in vitro* wear testing. The ISO Technical Specification No. 14596-2[17] describes millet seeds, poppy seeds and PMMA beads as food-simulating media. This selection was assumedly based on previously published wear tests, using these third-body media particles.[18-20] A recently published article reported that PMMA beads and natural seeds produce same order of volumetric wear and similar appearance of the worn composite surface micromorphology,[21] which is in striking contrast to our findings.

Inspection of the wear tracks revealed characteristic shapes, such as drop-shaped, tear-shaped, dumbbell-shaped, ellipsoid or reverse drop shaped. However no consistent correlation between the wear modes, two- or three-body loading, was detected. Therefore, the wear found both in terms of volume loss and in terms of wear track morphology reflected the interaction of the wearing modes and the differences in composition of the composite resins tested.

The morphology of DUR showed typical signs of fatigue-induced failures. Initially during two-body sliding this microfilled material with low Young's modulus is deformed under the opposing ball. The compression front is ahead of the sliding motion, thus plastic deformation produces a zone of tension behind. As a result, dissipation of the energy nucleates cracks, commonly perpendicular to the sliding direction. Thus, lateral cracking was detected along the boundaries of the prepolymerized particles, probably because the bond between such prepolymers and the surrounding matrix is weak. Under three-body loading with PMMA suspension deeper deterioration was noticed in all three segments of the track, presumably since loading of the PMMA beads had induced locally high stress concentration resulting in fatigue-like pitting.[11] Under the impact of the grinding edges of crushed poppy seeds and the load transferred by the ZrO_2-ball the surface of DUR was seriously damaged after 50,000 sliding cycles. Presumably, as a combined result of deformation, scratching and increased friction, debonding between prepolymers and matrix had occurred causing deep chipping fractures as also demonstrated with the high loss of volume. This observation is in line with the clinically reported catastrophic failure reported for microfilledocclusal restorations after several years in service.[22]

Much in contrast the hybrid composite APX showed high volume loss under water and PMMA suspension and very little volumetric loss during sliding in contact with poppy seed slurry. When in direct contact with the antagonist ball or the PMMA beads the largest fillers protruding from the worn surface are exposed to high stress and have therefore a tendency to be dislodged or exfoliated. This process is continuous as long as new large fillers are exposed. Under the grinding action of crushed poppy seeds the load is more uniformly distributed over the surface, meaning that the local stress on filler particles is reduced. The sharp edges of poppy seed fragments scratch and wear preferentially the inter-particle polymer. This phenomenon may be explained by the filler protection theory.[23,24]

Z250 and FIL showed approximately the same volume loss under each of the three wear tests. Monomer mixtures and filler loading of the products are very similar. The main difference is the kind and size of fillers, spherical particles of SiO_2 and ZrO_2 in Z250 and nanofiller clusters and discrete nanofillers in FIL. Under two-body wearing the hard spherical fillers of Z250 protruded from the surface and were continuously plucked-out during the course of repetitive sliding. Similarly, the clusters in FIL were preferentially worn, while the nanofiller particles in the surrounding polymer due to their size were apparently easily removed together with the polymer.[13] In the last third of the FIL tracks pronounced lateral cracking was seen, indicating fatigue-related wear. Sliding with PMMA slurry as third-body medium revealed characteristic patterns for both composites, albeit more pronounced for Z250. Alternating areas were seen with almost sound, non-abraded parts and areas clearly abraded revealing the structures of the materials. This phenomenon has been described as delamination and is presumably caused by surface and/or subsurface microcracks induced during plastic material deformation. When such cracks coalesce major cracks may be produced under the surface that finally may result in local loss of the wear surface, the delamination seen.[10,11] The effects of poppy seeds on both materials were superficial only, resulting in very low volume loss. Wear effects were most pronounced in the initial thirds of the tracks where the antagonist had touched the slurry in its normal composition, whereas the sliding ball might have pushed abrading seed particles ahead of the movement without further catching new slurry portions. The parallel shallow grinding scratches seen in the middle and terminal sections of the tracks support this assumption.

The morphological features detected along the sliding pathways for KAL and MFL are probably closely related to the low Young's moduli of these materials.[25] KAL with the large prepolymer particles showed compressed and delaminated zones under two-body wearing, MFL was in contrast uniformly worn along the track. PMMA slurry had produced high volume loss of substance, which is in agreement with the preferential loss of the matrix around the larger prepolymers of KAL and subsequent fracturing and exfoliation of the prepolymerized particles. In case of MFL the initial and middle wear morphology pictures from PMMA interaction with the surface showed multiple slabs. In the terminal third both materials displayed fractures and composite parts broken-out of the surface. Finally, when loaded under poppy seeds both materials showed severe cracking and deep fractures in the middle and terminal track areas, which is indicative for fatigue-failures.

The composite materials VED and VEP from the same manufacturer are identical in monomer composition. The bulk of the filler incorporated is silicate glass with an average grain size about 0.6 μm. The major difference is the addition of a small fraction of very large glass particles in VED and large non-filled prepolymers in VEP. It is a dominant finding, irrespective of the wear mode tested that the large glass fillers in VED protruding over the surrounding surfaces were loosened in the matrix or had been exfoliated leaving craters. In contrast the large prepolymers in VEP were abraded almost to the same level as the surroundings. Exposure to the PMMA slurry had produced large loss of volume. Under the action of the poppy seed slurry occasionally cracks could be seen in VED, mainly in the terminal section of the wear track, pointing on fatigue induced failure.

In summary, within the limitations of this laboratory study the following conclusions can be drawn: (1) Different wear modes produce different wear in terms of volume loss of substance and appearance of wear patterns. (2) Wear patterns produced as mutual interaction of composite, antagonist and wear medium are largely dependent on the composite resin composition, especially on kind, amount and size of the reinforcing filler used. (3) The morphological assessment of wear tracks reflects the nature of the abrasive and reveals insight into the mechanism generating wear patterns. (4) According to this *in vitro* study volume loss of composite resins is primarily caused by abrasive and fatigue-related wear mechanisms. (5) Selection of food-like slurries is required to simulate wear of composite restorations in occlusal cavities. (6) Isolated analyses of arbitrarily selected sites or of the deepest part of *in vitro* produced wear tracks can result in misleading interpretation of underlying wear mechanisms.

Comparisons of the *in vitro* produced wear patterns with the texture of occlusal restoration surfaces after several years in service would be highly desirable to validate *in vitro* test procedures.

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